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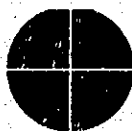
LSA LARGE AREA SILICON SHEET TASK CONTINUOUS LIQUID FEED CZOCHRALSKI GROWTH

(NASA-CR-158366) LSA LARGE AREA SILICON SHEET TASK CONTINUOUS LIQUID FEED CZOCHRALSKI GROWTH Quarterly Report, Oct. - Dec. 1978 (Siltec Corp., Menlo Park, Calif.)
28 p HC A03/MF A01 CSCL 13H G3/31 16667
N79-20281
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QUARTERLY REPORT

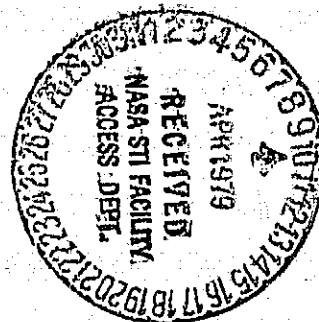
October - December, 1978

The JPL Low-Cost Silicon Solar Array Project is sponsored by the U.S. Department of Energy and forms part of the Solar Photovoltaic Conversion Program to initiate a major effort toward the development of low-cost solar arrays. This work was performed for the Jet Propulsion Laboratory, California Institute of Technology by agreement between NASA and DOE.



siltec

Menlo Park, California



January, 1979

LSA LARGE AREA SILICON SHEET TASK
CONTINUOUS LIQUID FEED CZOCHRALSKI GROWTH

QUARTERLY REPORT #5

October - December 1978

George Fiegl

January 1979

SILTEC CORPORATION

Menlo Park, California

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ABSTRACT

Siltec Corporation's contract with JPL is directed towards the design and development of equipment and processes, to demonstrate continuous growth of crystals by the Czochralski method, suitable for producing single silicon crystals for use in solar cells. Continuous growth is defined as the growth of 100 Kg of single silicon crystal, 10 cm in diameter, from one container. Siltec's approach to meeting this goal is to develop a furnace with continuous liquid replenishment of the growth crucible, accomplished by a melt-down system and a liquid transfer mechanism, with associated automatic feedback controls.

Elements of the transfer system were further developed and tested during actual transfer runs. Considerable simplification of the heating element of the transfer tube was achieved. Accuracy and reliability of the temperature sensor, which is part of the power input control system for the transfer tube, was improved. Electrical and thermal effectiveness were increased while assembly of the transfer tube system was further simplified.

Following the schedule of the growth demonstration plan, melt-down and transfer of molten silicon from the melt-down crucible into the growth crucible containing molten silicon was performed successfully, verifying the melt-down and transfer operation (rate of silicon transfer > 2 Kg/hr). After completion of this batch transfer, a crystal was grown from the transferred melt.

The final task of the growth demonstration plan prior to attempting 100 Kg throughput runs was achieved. This included the demonstration of continuous melt replenishment of the growth crucible while growing a crystal of 100 mm diameter and maintaining a constant melt level in the growth crucible. The proper replenishment rate and melt level constants of the growth crucible were achieved by raising the melt-down crucible at a velocity equal to the ratio of the solidification rate of the growing crystal and the melt-level area of the melt-down crucible.

Crystals grown from a melt with continuous liquid replenishment exhibited a very small or zero longitudinal resistivity gradient when the ratio of the dopant concentrations of the replenishing melt to the melt in the growth crucible was equal to the effective segregation coefficient of the used dopant.

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1.0 INTRODUCTION

The overall purpose of this program, which began in October 1977, is the demonstration of continuous crystal growth by the Czochralski method. Continuous is defined as a throughput of silicon that produces 100 Kg of single crystal material, 10 cm in diameter, from one common container.

The program includes:

- . Design and fabrication of a basic CZ furnace with a second chamber to be used for melting of the polysilicon.
- . Design, development, and fabrication of the continuous liquid silicon feed system with automatic feedback controls.
- . Demonstration of continuous crystal growth, as defined above, with an after growth yield >80%.
- . Definition of process control variables for continuous Czochralski growth.
- . Theoretical analysis of silicon sheet growth to be considered during design and development of the furnace.

The main effort during the last quarter was directed towards the development of the melt transfer system, and the demonstration of continuous liquid melt replenishment of the growth crucible, while growing a crystal, and maintaining constant melt level.

2.0 RESULTS

2.1 TRANSFER TUBE SYSTEM

The heating element for the transfer tube was further developed. A single heating element of the picket type, covering 50% of the quartz tube circumference, was used for all growth demonstration runs. The length of the unheated portion of the quartz transfer tube (heater end to melt surface) is critical to the avoidance of solidification of silicon in the tube when flow rates of $.24 \text{ cc sec}^{-1}$ are approached.

The maximum allowable length for our particular transfer system was determined to be $3/8"$. An increase of heater power output in the critical area of the tube end, a reduction of the unheated portion of the tube, and a decrease of the heat loss by conduction of the conical part of the vertical graphite tube eliminated the problem of melt solidification in this critical area which was experienced during some of our early runs.

A complete encapsulation with quartz of the thermocouple located near the transfer tube heating element, eliminated the problem of thermocouple wire breakage when reaching operating temperatures of 1450°C . A Platinum-Rhodium thermocouple type B was used for all testing.

The electrical insulation material (silica tape) was separated from the thermal insulation material by grafoil layers, allowing a reduction of the required thickness of the electrical insulation which would otherwise be penetrated by graphite felt fibers. The thermal insulation was made, to a large extent, an integral part of the outer graphite shell, simplifying the assembly of the transfer tube system considerably. Each of the

transfer tube assemblies is baked out under reduced pressure (30 Torr) at 750°C over 30 minutes to avoid contamination of the silicon melt through out-gasing, before an actual run is performed.

All materials used to fabricate the transfer system are materials that have been in use for many years in standard CZ furnaces. (See Appendix I pages 12-15.)

2.2 MELT-DOWN AND TRANSFER OF MOLTEN SILICON

As part of the growth demonstration plan, melt-down and transfer of liquid silicon from the melt-down chamber into the growth crucible containing molten silicon was performed. Both the melt-down and the growth crucibles were filled with 7.5 Kg of poly crystalline material. The melting process was started simultaneously in both systems. During melt-down the temperature of the transfer tube was brought up to 1450°C (indicated by a Pt-Rh thermocouple located close to the quartz transfer tube). After completion of the melt process in both chambers, the melt in the growth crucible was brought to seed dip temperature, while the melt in the melt-down side was kept approximately 50°C above dip temperature; this was done to eliminate the possibility of solidification of the melt inside the transfer tube when first filled with molten silicon by pressure differential.

After temperature stabilization of the melt in both systems, both tube ends are submerged into the melt by raising the crucibles so that the unheated tube portion between tube heater and melt level is not more than 3/8" in length. By applying a pressure differential (35 in. H₂O) between the two chambers, the tube is filled. Generating the pressure is accomplished by closing the argon exhaust valve on the melt-down side, and the pressure equalizing valve between the two chambers. Both valves are manually operated and are opened immediately when a small melt level drop in the melt-down chamber is detected. Melt can now be transferred at controlled rates through a pre-determined head differential. Melt transfer can be accomplished in both directions.

Melt transfer was achieved at rates > 2 Kg/min.
Crystals were grown from melts after replenishing
with several Kg of melt. No negative effects were
experienced during the growth of these crystals.

2.3 CONTINUOUS REPLISHMENT WHILE GROWING A CRYSTAL

Continuous melt replenishment of the growth crucible was accomplished during simultaneous growth of a crystal while the melt level in the growth crucible was kept constant.

After the tube was filled with liquid silicon as described in the first paragraph, "Melt-down and Transfer of Molten Silicon" standard CZ procedures such as "necking" and "shouldering" were followed. During growth of the cylindrical section of the crystal, the melt-down crucible was raised at a velocity equal to the ratio of the solidification rate of the growing crystal to the melt level area of the melt-down crucible. Practically this was achieved by "slaving" the crucible lift mechanism electrically to the seed pull mechanism, using the appropriate ratio of crystal to crucible diameters. By applying this procedure, the melt level remained constant (indicated by the laser melt level control system).

A correlation between hydrostatic pressure and replenishment rate for a given tube diameter is derived from Bernoulli's equation when operating below critical Reynold's numbers. (See Appendix I, pages 16 and 17.) The temperature of the transfer tube was kept at 1450°C during continuous replenishment by means of a 1.5 KW power input.

During the first continuous replenishment run approximately 5Kg of melt were transferred while growing a 4" diameter crystal.

When the melt level in the melt-down crucible dropped too low to allow further melt transfer, the lift

mechanism of the growth crucible was electrically "slaved" to the seed pull mechanism, while the melt-down crucible lift was stopped. This procedure will be applied when pulling the last crystal of multiple ingot runs. The growth of the crystal was not disturbed by the continuous replenishment of the melt. (See Appendix I page 18.)

2.4 UNIFORM LONGITUDINAL IMPURITY DISTRIBUTION

Crystals grown with continuous liquid replenishment exhibited a small, or zero, longitudinal resistivity gradient when the ratio of the dopant concentrations of the replenishing melt to the growth crucible melt is kept constant and is equal to the effective segregation coefficient of the dopant used.

Practically, this is achieved by doping the initial melt of the growth crucible for the desired resistivity target of the ingot, taking the effective segregation coefficient into account. The dopant concentration in the melt-down crucible is lower and equal to the dopant concentration of the solid (growing crystal).

3.0 CONCLUSIONS

- 3.1 A single heating element of the picket type formed from grafoil strips is suitable for achieving a uniform temperature distribution along a quartz transfer tube; so that during transfer of molten silicon at a rate of 0.24 cc Sec^{-1} no solidification in the tube occurs.
- 3.2 Continuous liquid replenishment of the growth crucible, while growing a crystal and keeping the melt level constant can be accomplished through a siphon, after initial filling of the transfer tube by pressure differential, when the melt-down crucible is raised at a velocity equal to the ratio of the solidification rate of the growing crystal to the melt level surface area of the melt-down side.
- 3.3 The longitudinal resistivity gradient of a crystal grown from a melt with continuous liquid replenishment is small or zero when the ratio of the dopant concentrations of the replenishing melt to the growth crucible melt is equal to the effective segregation coefficient of the dopant used.

4.0 PROJECTION OF ACTIVITIES

During the remaining three months of the contract, three one-hundred Kg throughput runs are scheduled for the purpose of establishing economic data.

One run is planned for January, and the other two runs are scheduled for February.

DRL No. 61/DRD No. SE/Line Item No. 7

DOE/JPL-954886-79/1
Distribution Category UC-63

APPENDIX I

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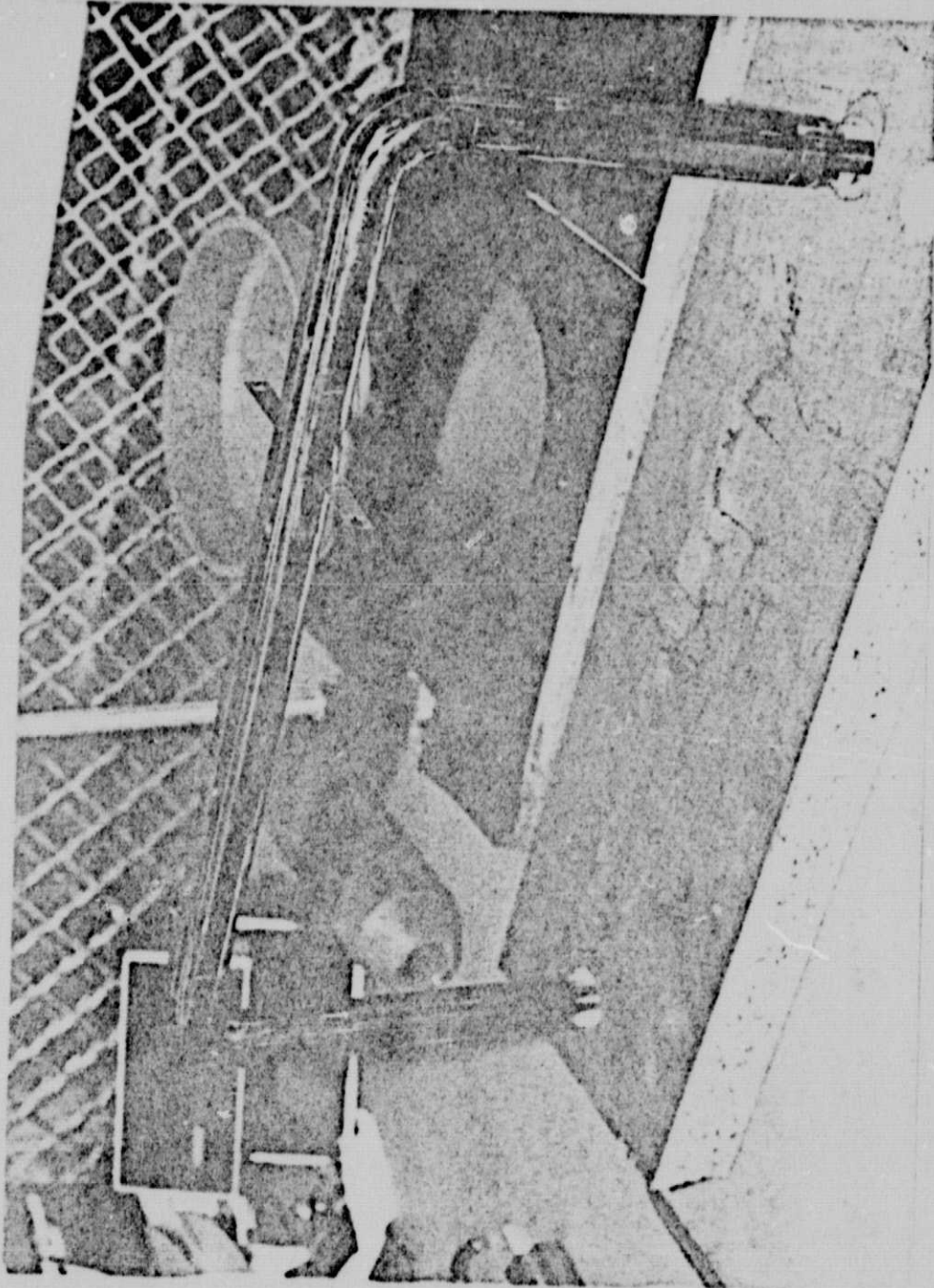


FIGURE 1. QUARTZ TRANSFER TUBE WITH ATTACHED GRAFOIL STRIP
HEATER AND MOLYBDENUM ELECTRODES

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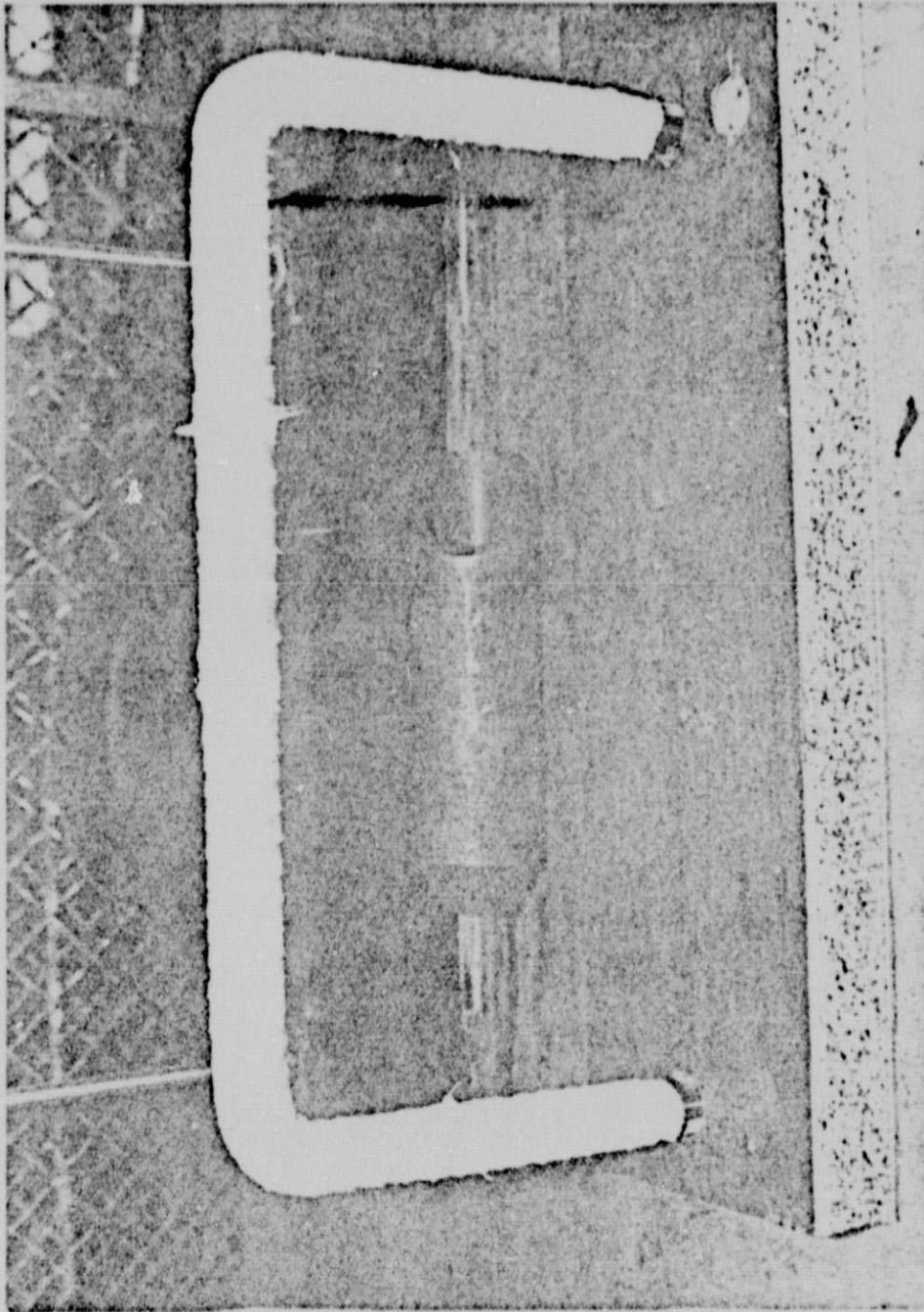


FIGURE 2. TRANSFER TUBE SURROUNDED BY SILICA TAPE LAYER
FOR ELECTRICAL INSULATION

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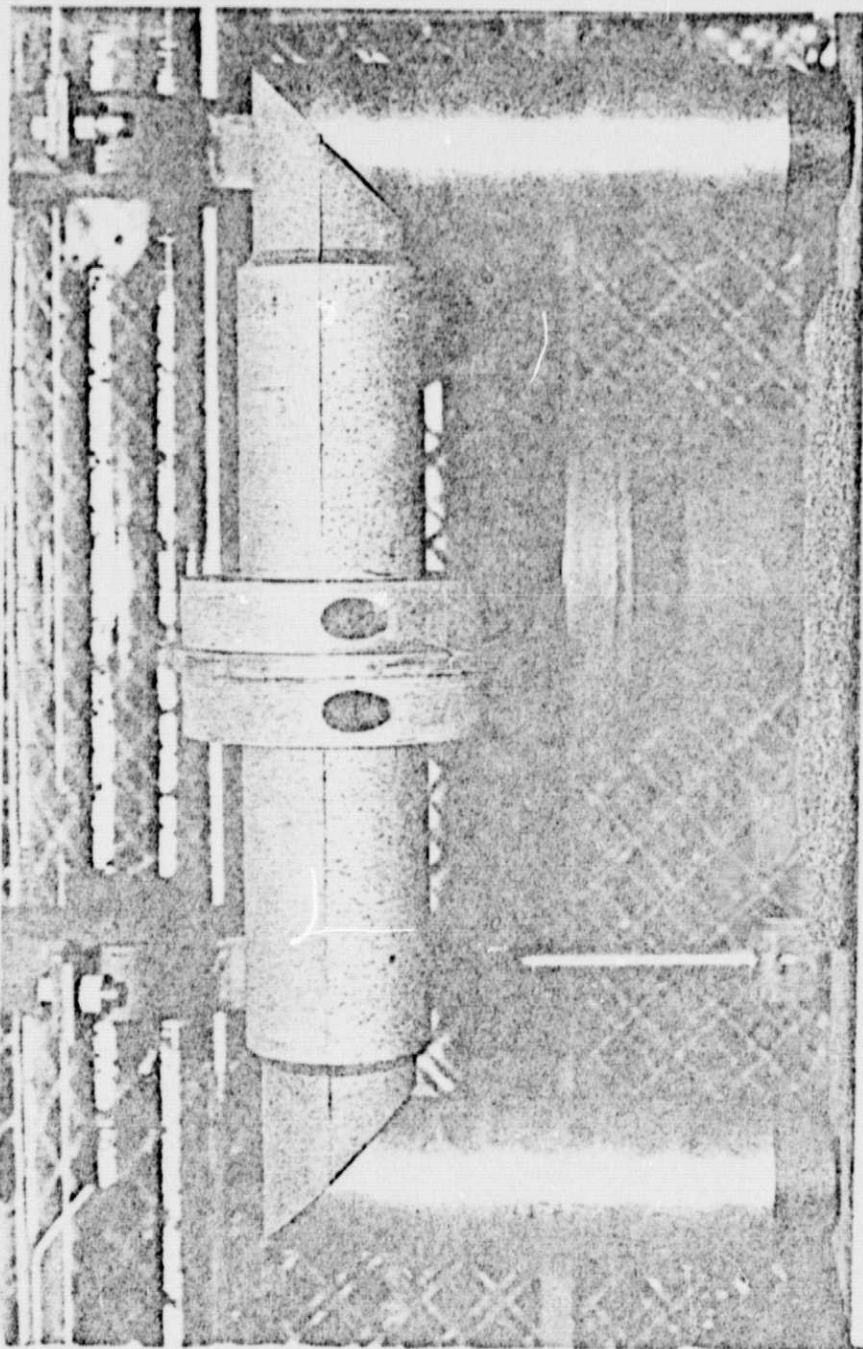


FIGURE 3. COMPLETELY ASSEMBLED TRANSFER TUBE WITH MOUNTING
FLANGE AND OUTER GRAPHITE SHELL

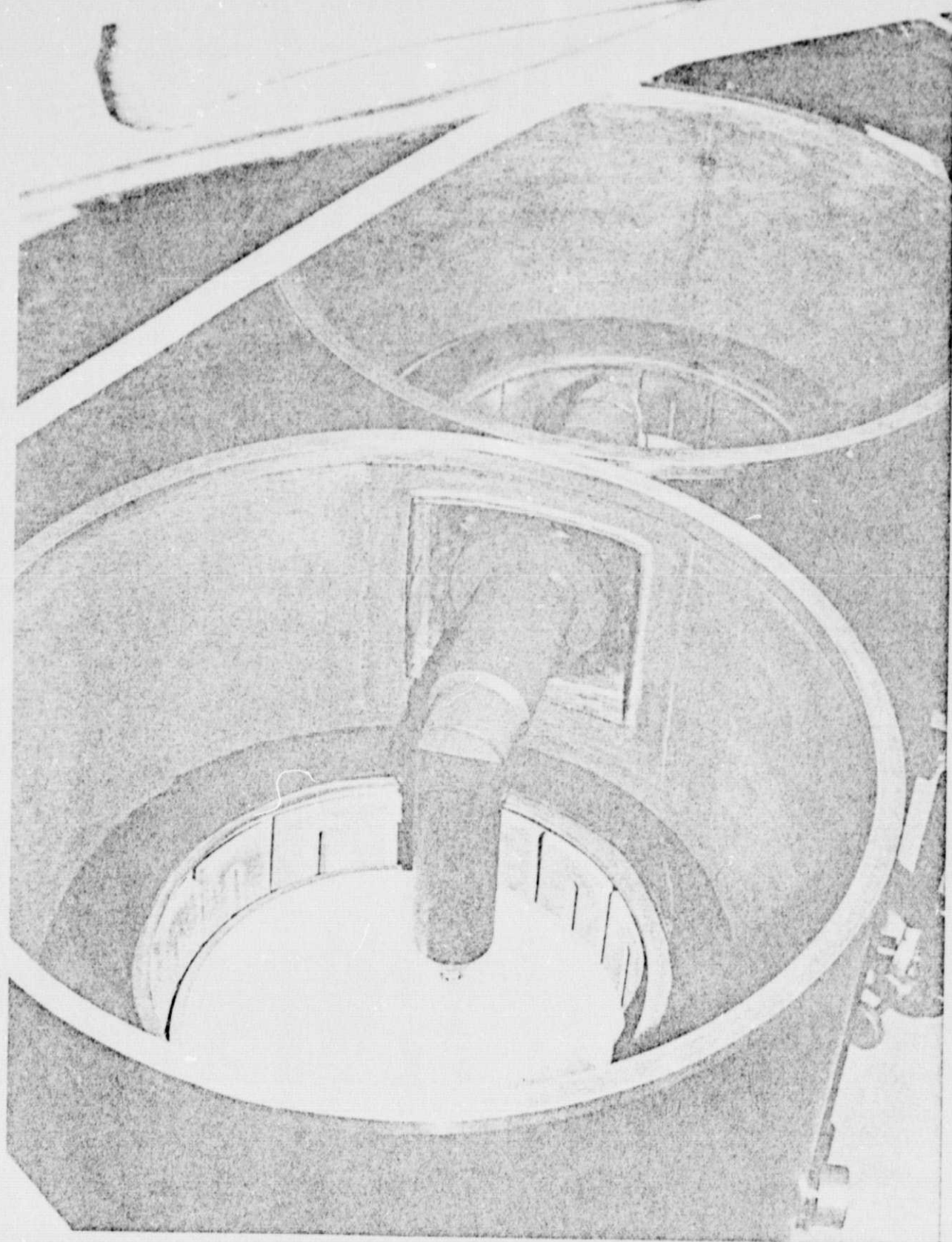
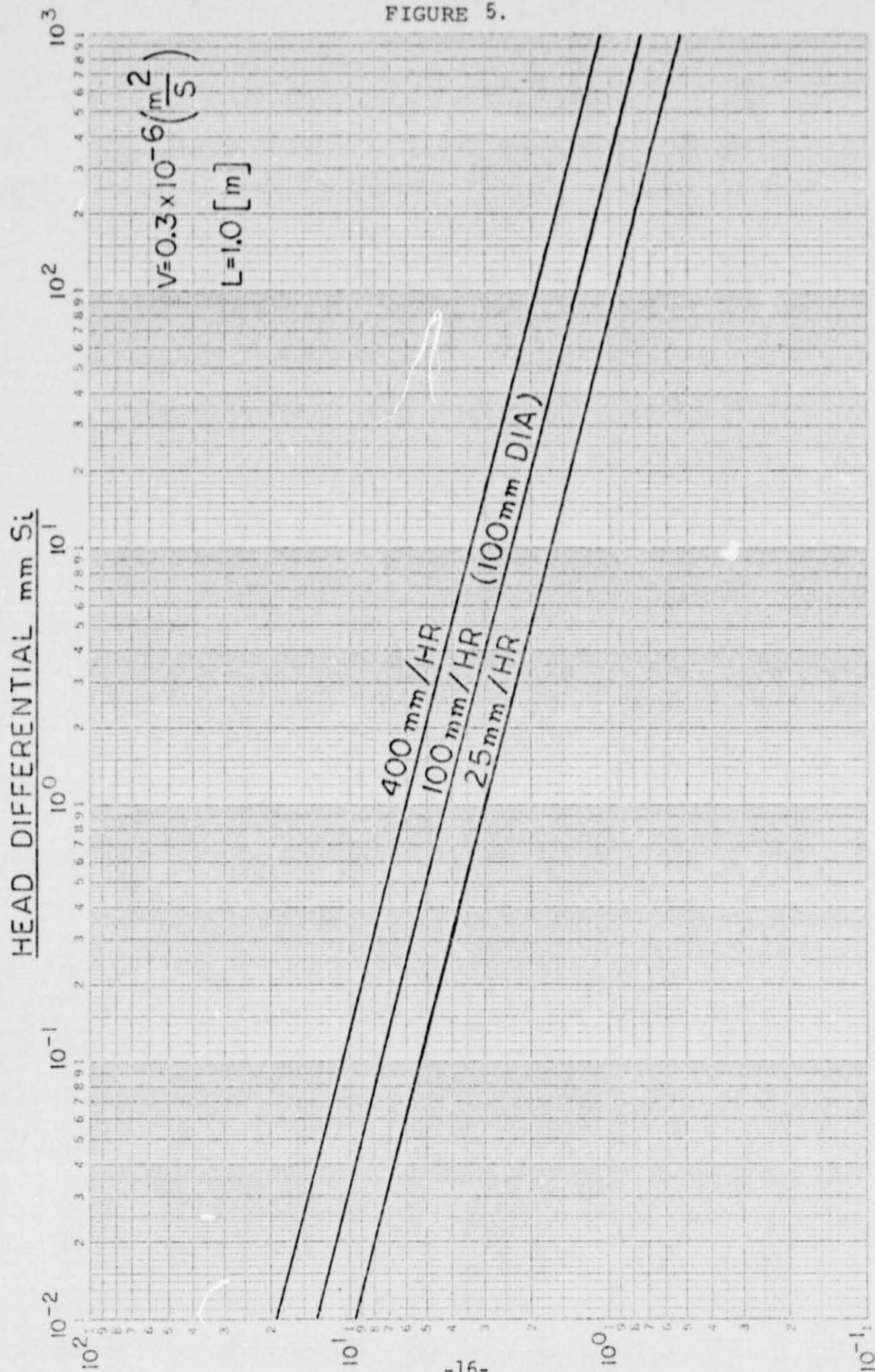


FIGURE 4. INSTALLED TRANSFER TUBE SYSTEM

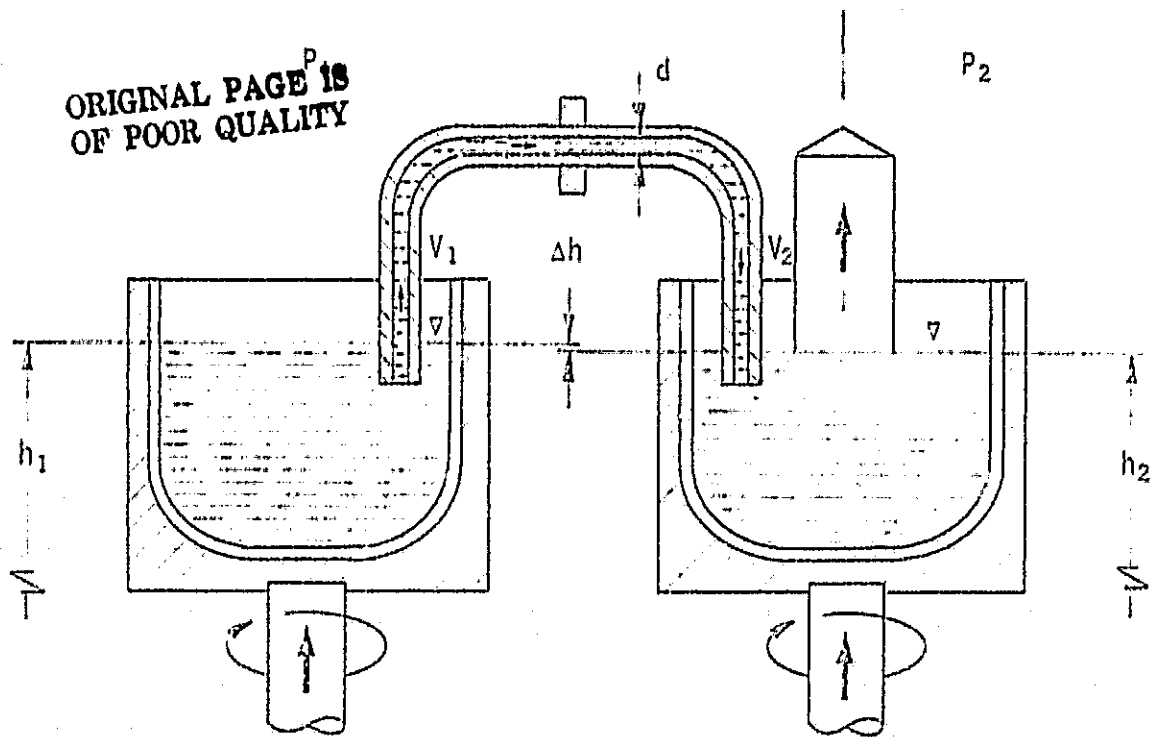
TUBE I.D. mm

FIGURE 5.



HEAD DIFFERENTIAL AS A FUNCTION OF
 REPLENISHMENT RATE AND TUBE I.D.

FIGURE 6.



$$(1) \quad \frac{V_1^2}{2g} + \frac{P_1}{\gamma} + h_1 = \frac{V_2^2}{2g} + \frac{P_2}{\gamma} + h_2 + \xi \frac{V_2^2}{2g}$$

$$V_1 = V_2$$

$$P_1 = P_2 \text{ for CLF}$$

$$(2) \quad \text{If } \frac{Q}{d} \cong 5.6 \times 10^{-4} \frac{\text{m}^2}{\text{s}} \Rightarrow \Delta h = \frac{128 L \cdot v \cdot Q}{d^4 g \cdot \pi}$$

Δh	head differential	{m}
L	tube length	{m}
v	kinematic viscosity	$\{\frac{\text{m}^2}{\text{s}}\}$
Q	replenishment rate	$\{\frac{\text{m}^3}{\text{s}}\}$
d	I.D. of transfer tube	{m}

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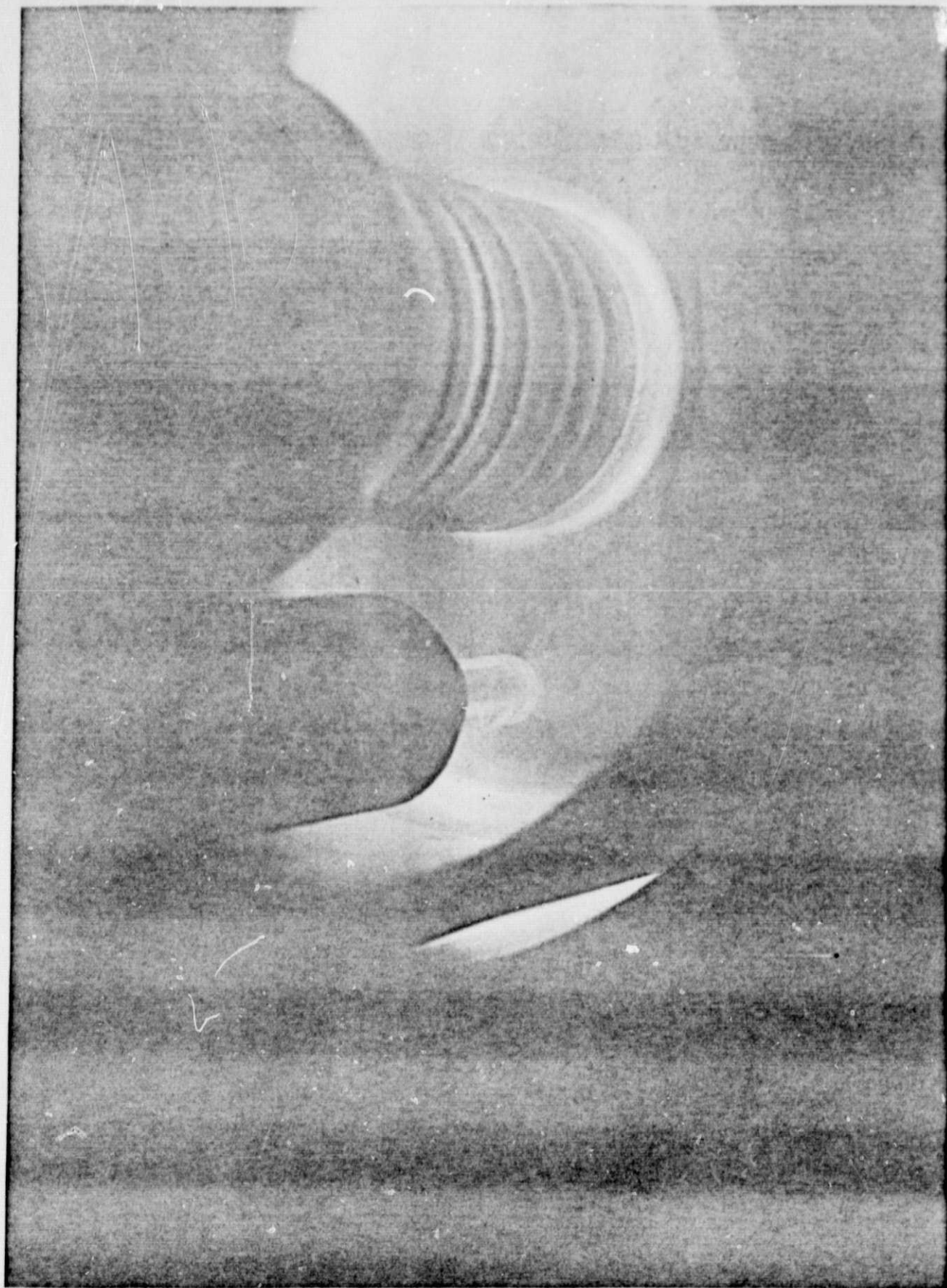


FIGURE 7. MELT IS CONTINUOUSLY REPLENISHED WHILE GROWING A CRYSTAL

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APPENDIX II
NEW TECHNOLOGY

No new technology was generated during the reporting period.

APPENDIX III

Summary of Characterization, Design, and Economic Analysis Data

No new data were generated during this quarter to analyze.

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APPENDIX IV

Milestones

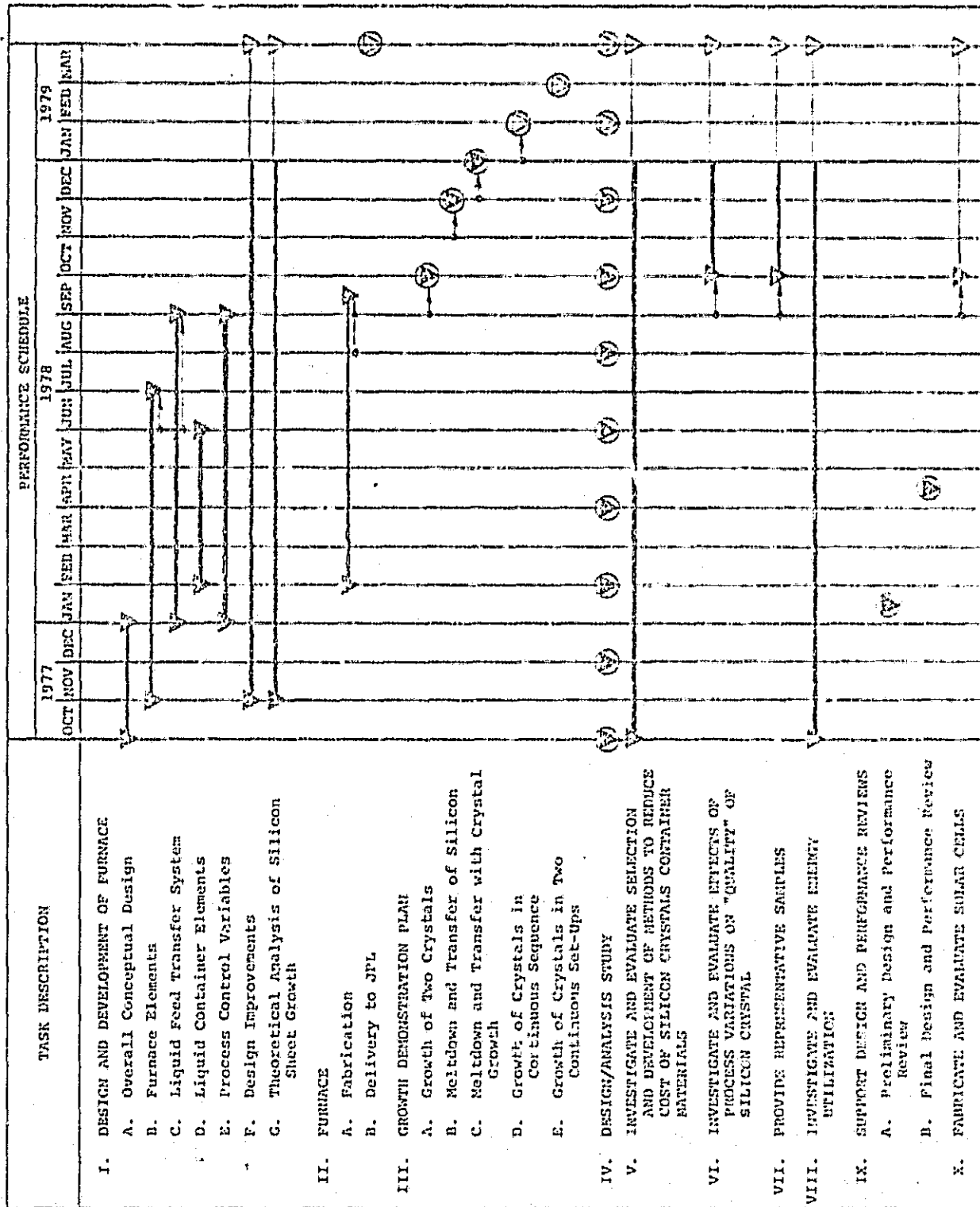
Labor Summary

Cost Summary

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PROGRAM PLAN

Revised: 6-8-70



Milestone Legend:

○ = Data Taken
□ = Start/Finish
△ = Program Division